

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

U.S. Patent Application No.:	09/998,469)	
Filing Date:	November 29, 2001)	Group Art Unit: 2814
For:	Barrier Layers For Protecting Metal Oxides From Hydrogen Degradation)	Examiner: Peralta, Ginette
Applicants:	Solayappan et al.)	Docket No.: 13176.403
)	Confirmation No.: 5686
)	Attachment to Paper No.: 9

CERTIFICATE OF TRANSMISSION UNDER 37 CFR 1.8

I hereby certify that this correspondence, along with all papers referred to as being transmitted, are being facsimile transmitted to the Patent and Trademark Office Fax No. (703) 308-7724.

September 16, 2003
Date

Elaine C. Von Spreckelsen
Elaine C. Von Spreckelsen

DECLARATION UNDER RULE 131

We, Narayan Solayappan, Jolanta Celinska, Vikram Joshi, Carlos A. Paz de Araujo and Larry D. McMillan, hereby declare:

1. We are the inventors of the above-identified patent application.
2. Attached as Exhibit A is a copy of an invention disclosure for the above-identified patent application.
3. This invention disclosure is a true and correct copy of a document prepared by one of us (Narayan Solayappan) on or about August 1, 2001, and sent to patent attorney Carl A. Forest of Patton Boggs LLP.
4. The invention disclosure substantially discloses all elements of independent claims 1, 28, 38 and 68 of the above-identified patent application.
5. As indicated by the table and figures which are part of the invention disclosure, actual hydrogen barrier layers as described and claimed in the present application were fabricated and tested before August 1, 2001.
6. The samples disclosed in the Invention Disclosure and discussed above

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were made and tested in Colorado Springs, Colorado, USA.

7. Thus, the invention as claimed was reduced to practice in this country by August 1, 2001.

8. As shown by the subject line of Exhibit A, which reads "Patent Application for a new hydrogen barrier", the disclosure was received by our attorneys Patton Boggs LLP, on or about August 1, 2001.

9. Thus, we had done everything we could to initiate the filing of a patent application on this invention prior to the effective date of September 10, 2001, of the Kanaya reference, US2002/0038402 A1.

10. As indicated by the record in this application, the present application, Patent Application Serial No. 10/998,469 was filed on November 29, 2001.

11. We hereby declare that all statements made herein of our own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like are punishable by fine or imprisonment, or both, under 18 U.S.C. §1001, and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

September 11, 2003
Date

Narayan Solayappan
Narayan Solayappan

September 11, 2003
Date

Vikram Joshi
Vikram Joshi

09/11/03
Date

Jolanta Celinska
Jolanta Celinska

Sept. 11, 2003

Date

CF-FLH

Carlos A. Paz de Araujo

Sept. 11, 2003

Date

Larry D. McMillan

Larry D. McMillan

Date: August 1st, 2001
From : Narayan Solayappan
To: Carl Forest
Subject: Patent Application for a new hydrogen barrier

AUTHORS: Narayan Solayappan, Jolanta Celinska, Vikram Joshi, Carlos Araujo and Larry McMillan

As I mentioned to you over the phone, please find the following information on the new hydrogen barrier.

The new hydrogen barrier is Strontium Tantalate (STO) (SrTa_2O_6) deposited by MOCVD technique. The deposition conditions are shown in Table 1. Please see Figure 1 and 2 for hysteresis and leakage data of SBT films measured before and after forming gas anneal (FGA). STO was deposited as a hydrogen barrier on top of the SBT wafers prior to FGA.

As deposited STO films are amorphous and very dense as revealed by X-ray diffraction data and SEM surface analysis. It is believed that the amorphous dense nature which helps STO to act as a hydrogen barrier. Such amorphous dense films can be obtained at low deposition temperatures ($\sim 450^\circ\text{C}$) easily by a deposition technique like MOCVD. Spin-on/LSMCD techniques (annealed at such low temperatures) would give leaky films and moreover does not result in dense films. We are currently evaluating the use of Tantalum penta-oxide (TO) and Bismuth Tantalate (BTO) as hydrogen barriers deposited by MOCVD. The patent should cover STO, TO and BTO materials deposited by MOCVD for hydrogen barrier application.

Figure 3 shows the CV plot of as deposited MOCVD-STO and MOCVD-STO annealed at 650°C in oxygen ambient for a total of 1 hour. The dielectric constant of both as-deposited ST and annealed ST are very similar indicating that the material is amorphous even after a $650^\circ\text{C}/1\text{ hr}$ anneal and there is no grain growth during the anneal (this is

further evident from XRD and SEM data). This is an important requirement for a hydrogen barrier due to the fact that any recovery anneals performed for SBT in a process flow does not change the amorphous nature of STO and hence barrier properties of STO are maintained.

The patent application should also cover the use of these hydrogen barriers as double layers. One layer would be deposited after TE patterning of the SBT ferroelectric capacitor. This layer would be exposed to high temperature during recovery anneals of SBT. The other layer would be deposited after Metal 1 deposition. After Metal 1 (Aluminum) deposition in a CMOS process flow, the wafers cannot be exposed to temperatures greater than 450 °C. Thus, low temperature (≤ 450 °C) as-deposited MOCVD-ST can serve as a hydrogen barrier after Metal 1 deposition as well.

We will keep you informed as we make further progress regarding this subject.

You can call me if you have any questions.

Thanks,

Narayan Solayappan.

Deposition Temperature	~450 °C
Pressure	3 mbars
Oxygen flow	600-800 ccm
Argon flow (carrier gas)	200-300 ccm
Source	ST modified double alkoxide in toluene solvent, 0.05M (ST penta ethoxide - 2-methoxy ethoxide)
Liquid flow	0.2-0.3 ccm
Film thickness	600-800 Å

Table 1. Deposition Conditions of MOCVD ST

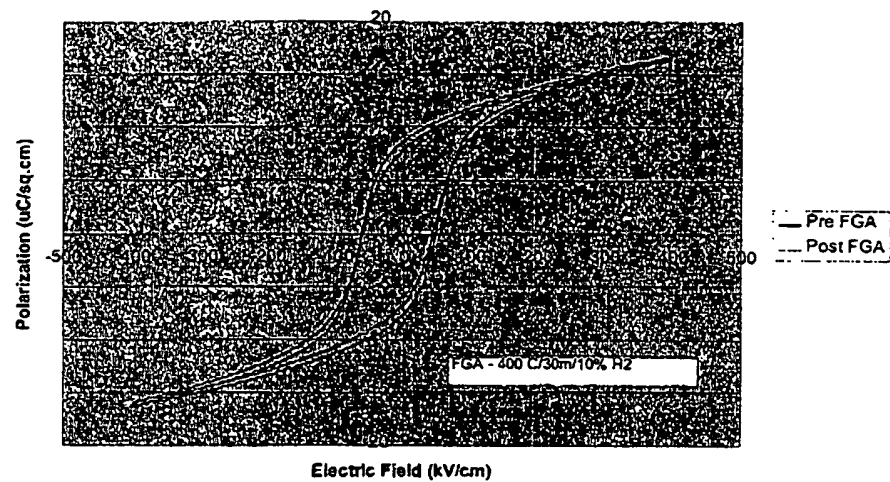


Figure 1. Hysteresis data before and after FGA

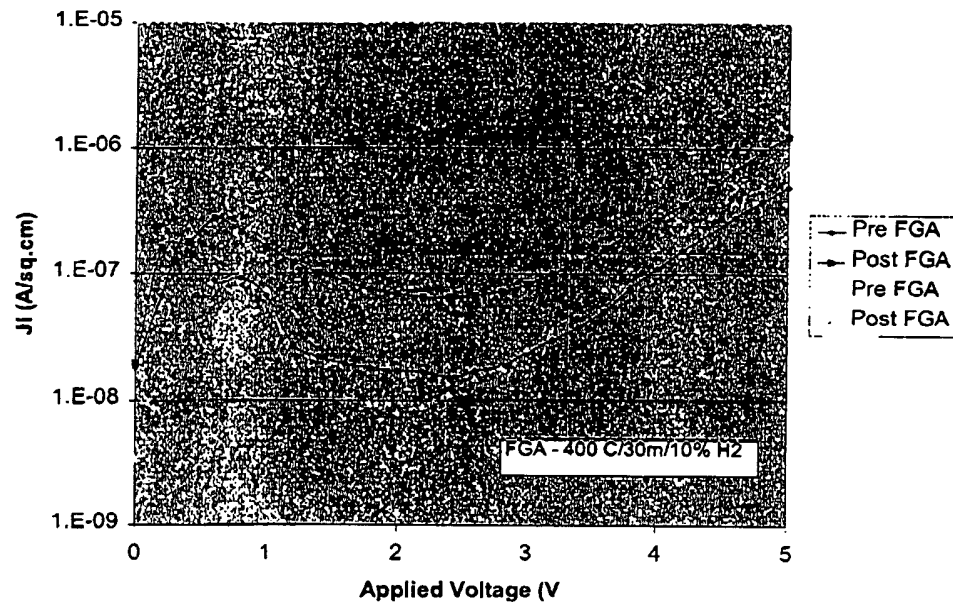
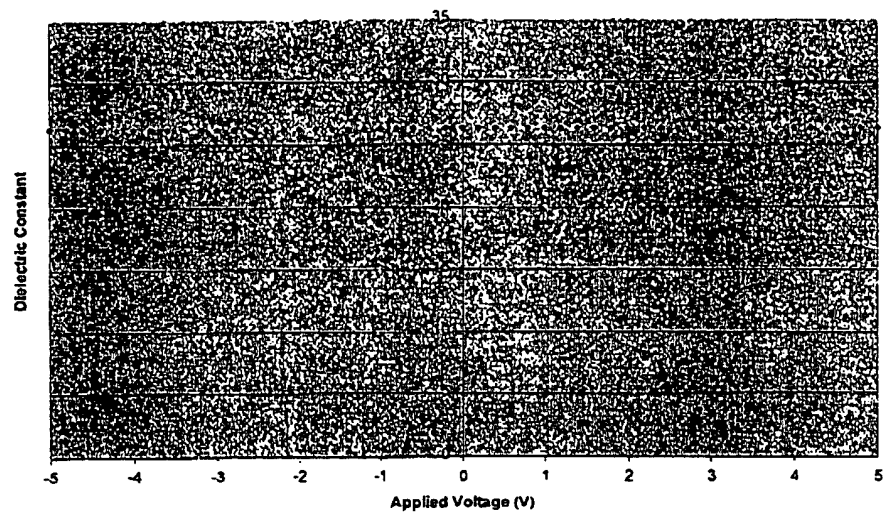


Figure 2. Leakage data before and after FGA

C-V Plot of as deposited MOCVD S



C-V Plot of MOCVD-ST annealed at 650 C /O2 for 1h

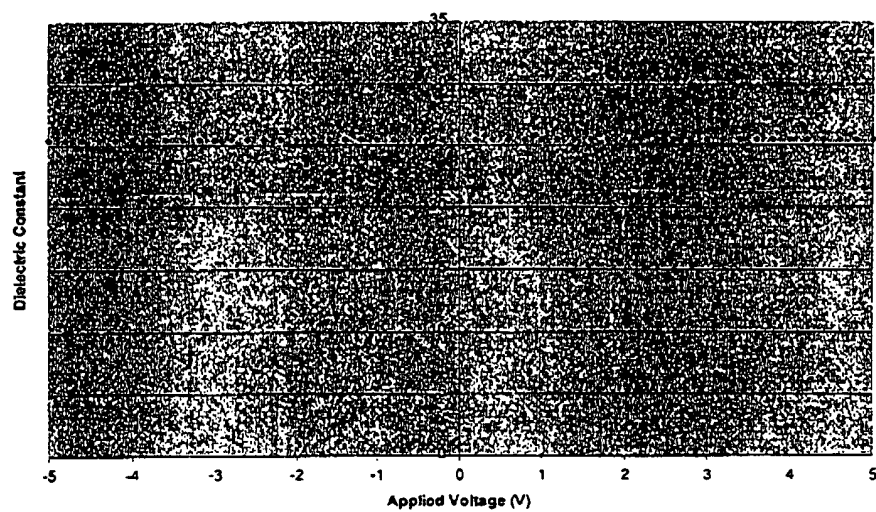


Figure 3. CV Plot of as deposited and annealed ST capacitors